

## Technical Note SCD 2

# Design Principles for Modern Multilayer Optics

The design and technology of X-ray optics has progressed very rapidly over the past several years. State-of-the-art multilayer optics, when coupled to high brightness microfocus rotating anodes, are now capable of producing beams that rival second generation synchrotron sources in intensity.

Bruker AXS invented and introduced the first modern multilayer optics in 1995 (the R&D 100 award winning Goebel optics).

Here we describe the design principles of modern multilayer optics and the unique features of Bruker Helios optics.

### Principles of Multilayer Optics

A multilayer optic is essentially an artificial, two dimensional crystal. The spacing of the multilayers is designed so that, at each point on the mirror, Bragg's diffraction law is satisfied

$$\lambda = 2d \sin \theta$$

where  $\lambda$  is the wavelength of the characteristic emission of the source,  $d$  is the spacing of the multilayers and  $\theta$  is the diffraction angle. The optic is shaped, and the spacing of the multilayers is graded such that the characteristic radiation is in Bragg resonance at each point on the mirror and focuses to a single point. Other wavelengths do not

satisfy the conditions for Bragg reflection and are thus not reflected, thus allowing the mirror to produce a highly monochromatic beam.

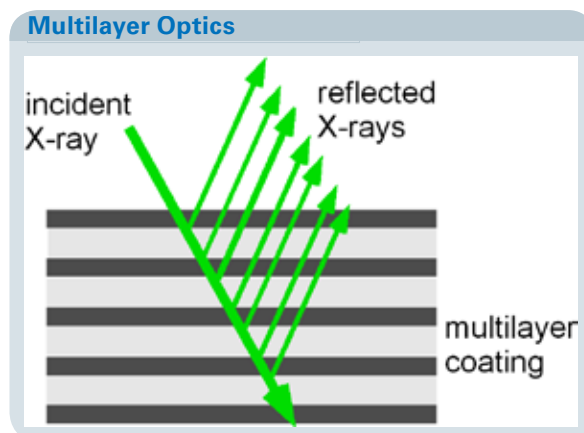


Figure 1: Operating principle of multilayer optics. X-rays are reflected by diffraction at a series of interfaces between layers with differing indices of refraction. Only the wavelength of interest is effectively reflected, other wavelengths are absorbed in the mirror, producing a monochromatic beam.

The optic is typically designed as either a single, doubly curved optic (single bounce) mirror or a two-mirror (double bounce optic). Examples are shown in Figure 2. Single bounce optics have the theoretical advantage of higher reflectivity compared to double bounce optics.

The polishing required to shape the substrate however, results in roughness that reduces the multilayer reflectivity of the single bounce optic. Furthermore, because there is only a single bounce, the spectral purity of this type of optic is several orders in magnitude less than multi-bounce mirror designs. Because of this, double bounce optics are typically recommended for protein crystallography.

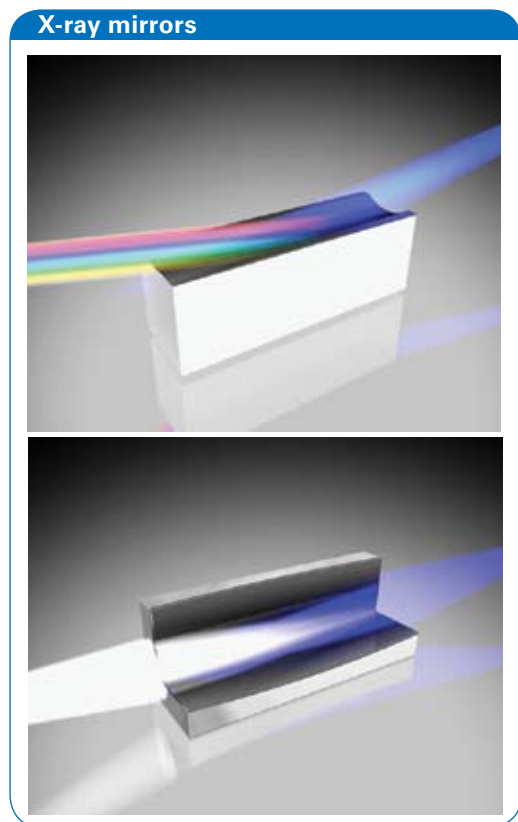


Figure 2: Examples of a single bounce X-ray mirror (left) and a double bounce mirror (right). Both mirrors focus the incident X-rays at the sample location. Double bounce mirrors produce a much purer spectrum and are thus preferred for protein crystallography.

### Beam Size

Ideally the size of the beam at the sample should be comparable to the size of the sample. If the beam is much larger than the sample, then most of the X-rays will simply miss the sample as shown in Figure 3 and are thus wasted. Worse, these wasted X-rays actually increase the scattered X-ray background which degrades the signal-to-noise ratio.

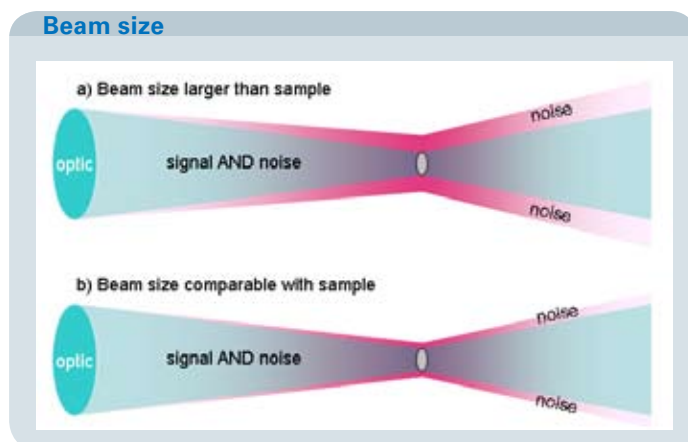


Figure 3: A high flux optic is not desirable for smaller samples if most of the flux misses the sample. (b) A higher intensity optic concentrates the flux on the small sample improving the signal-to-noise ratio.

If the beam is very much smaller than the sample, then X-ray absorption errors are enhanced (since the volume of the intersection of the crystal and the beam will vary more than if the crystal is completely illuminated).

Pinholes or collimators are used to reduce the beam size at the sample and to reduce the noise. They can however only remove some of the excess flux, not all of it, as illustrated in Figure 4. Thus, also when using pinholes to shape the beam, the best results are obtained using a beam with a size comparable to that of the sample size.

Since most crystals of interest for macromolecular crystallography are on the order of 100-200 microns, the Helios optic is designed to produce a beam at the sample of slightly more than 200 microns. Competing optics produce beams that are significantly larger, thus producing more noise.

	VariMax	Helios	Comments
Maximum intensity gain (relative to Osmic "Blue" optics)	2.5	6.4	Helios provides more than twice the intensity gain of the Varimax. More photons on sample: <b>shorter measurement time.</b>
Beam size	0.38	0.21	Helios beam better matched to size of typical protein crystals. Less photons miss sample: <b>better S/N ratio.</b>
Maximum unit cell axis (Å)	450	450	
Beam shape	square	circular	Ideal circular beam shape allows <b>better integration</b> quality and <b>higher resolution</b>
Radiation protection	helium purge	vacuum	<b>Vacuum purge</b> does not run out like helium

Table I: Comparison of Helios and Varimax optics.

In short, the Helios' performance is characterized by:

- more flux on the sample -> shorter measurement time
- less flux past the sample -> better signal-to-noise ratio

Finally one remark about the difference between convergent angle and capture angle. The difference is illustrated in Figure 9. Convergent angle is the solid angle put onto the sample, and directly relates to the intensity at the sample. The capture angle is the solid angle taken from the source, and is related to the total flux that is in the beam. A large capture angle is not always good. For instance, a large solid angle can be made by placing the optic close to the source (small  $f_1$ ). Indeed, the total flux in the beam increases, but at the same time the beam size  $D$  at the sample increases as well (see above equations). The amount of photons that miss the sample is thus increased, not the amount of photons on the sample.

### Prevention of Radiation Damage

It is well known that multilayer mirrors can be irreversibly damaged by exposure to the ozone which is produced by the X-ray flux through the optic. This can happen quite rapidly (in a matter of hours) on modern high intensity rotating anodes. Because of this, modern optics are typically purged to remove all oxygen.

Often the optics are purged with helium. This both protects the optic from damage and also reduces air absorption. For example, the VariMax optics employ a helium purge.

However, the helium purge requires periodic changes of the helium canister. Failure to replace the canister can lead to damage of the optics since there is no interlock which prevents the source from operating without helium.

Because of this, Bruker Helios optics employ a vacuum purge. This also reduces air scatter and protects the optic. However, unlike a helium bottle which needs to be manually replaced periodically, the vacuum pumps works continuously, never requiring manual maintenance. In addition, if for any reason the vacuum is lost, a interlock will close the shutter of the generator in order to prevent damage to the optics.

### Summary

The Helios optics are the most advanced multilayer mirrors available. They feature the highest intensity gain and their true circular beam shape allows better spot separation and better profile fits. They provide a beam size designed to match the typical size of macro molecular crystals for the best signal-to-noise ratio. Their innovative vacuum purge never requires user maintenance (unlike other optics which require a periodic exchange of helium bottles).

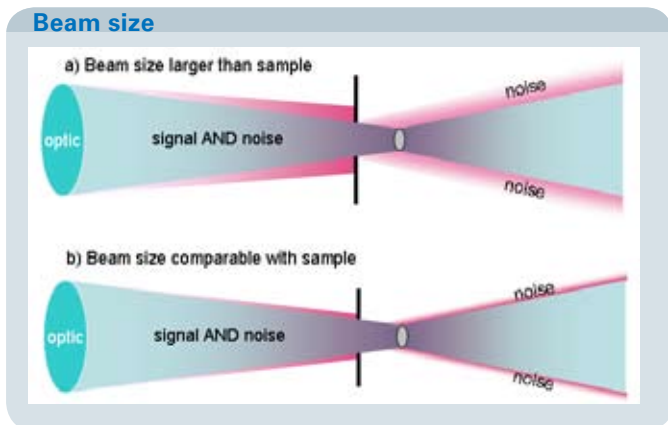


Figure 4: A pinhole or collimator can remove some of the flux that misses the sample, but it can not completely remove it. Also with a pinhole the best signal-to-noise ratio is obtained with a beam that has a comparable size as the sample.

### Spot Shape

The ideal shape of the diffracted spot is circular. Most integration programs assume circular (or elliptical) spots and thus ideal circular spots give the best peak fitting and thus the best data quality. However, most multilayer optics do not produce an ideal circular beam but rather a beam which is a tilted square (diamond-shaped) in cross section. This is schematically illustrated in Figure 5 (below). This non-ideal square profile is due to both the rectangular geometry of the optics and the crossed slits used to control the beam divergence.

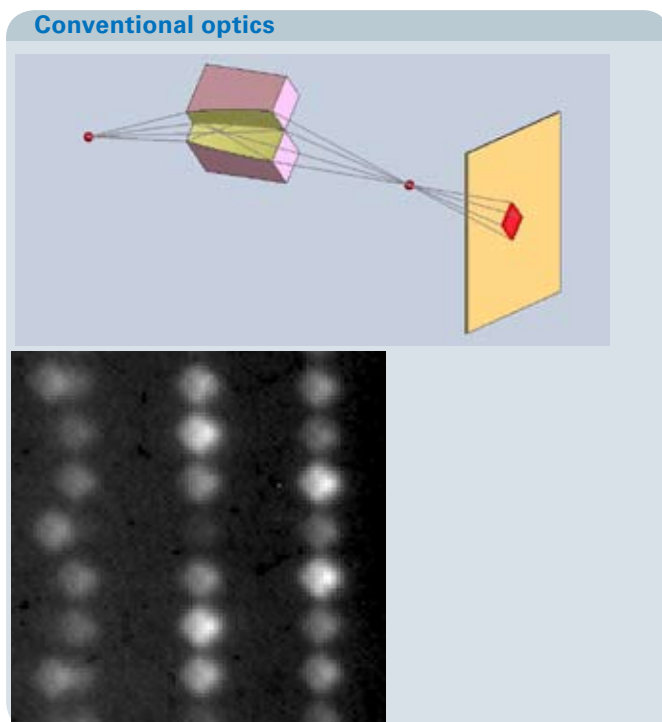


Figure 5: The beam from a conventional double bounce mirror is square in cross section (upper). The diffracted beams are therefore also squarely shaped (lower).

A rectangular beam cross section is not ideal. The diffracted beam is a convolution of the beam divergence with the crystal shape and mosaicity. The resulting diffracted beams will thus also tend to be square, as shown in Figure 5.

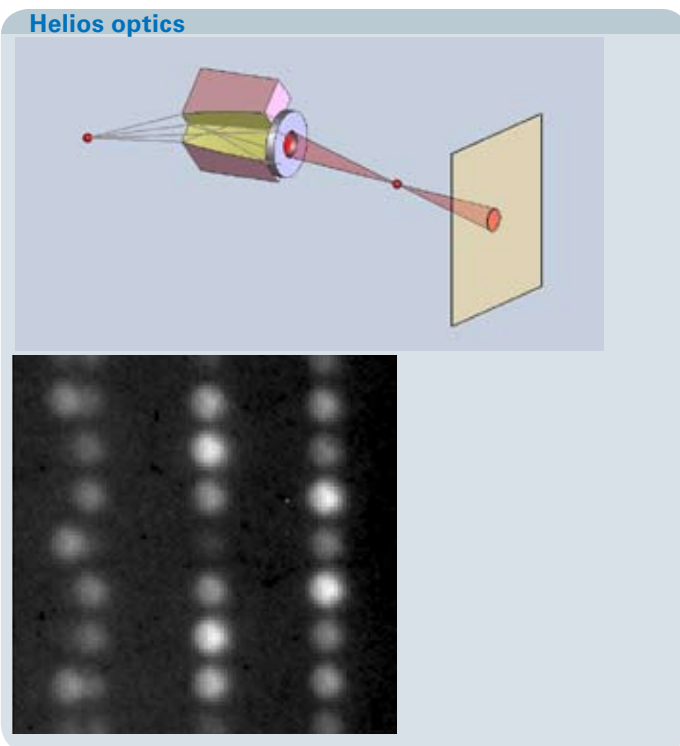


Figure 6: Helios optics in Bruker diffraction equipment use circular apertures (upper). The resulting circular beam produces circularly shaped diffraction spots (lower), which are the least prone to overlap.

Since square reflections overlap more readily than circular reflections, Helios optics in Bruker diffraction equipment use pre-set apertures to produce the ideal circular beam. See Figure 6. It is the only system available that produces an ideal circular beam (rather than the square beam produced by other multilayer optics).

## Flux and Intensity

Flux is the total number of X-ray photons per second focused by the optic. Intensity is the number of photons per second per unit area. It is often assumed that flux is the most important characteristic of the mirror performance. However, as noted above, flux that does not hit the sample is wasted in that it does not contribute to the diffracted signal and, worse, it increases the scattered X-ray background (which thus further degrades the signal-to-noise). Therefore, a mirror with high flux may have very poor performance if its beam size is too large.

Because of this, intensity (also known as flux density) is a much more accurate predictor of mirror performance. A mirror with high intensity will deposit more X-rays onto the sample (and the diffracted signal is directly proportional to the intensity times the crystal size).

The intensity of the system is given by the well-known Liouville's theorem:

$$I=BR\Omega$$

Where B is the brightness of the source (in photons/mm<sup>2</sup>-sec-steradian), R is the reflectivity of the optic and  $\Omega$  is the convergence solid angle of the optic (in steradians). For a given X-ray source, the brightness is a property only of the source and can not be changed by the optic. The mirror reflectivity is determined by the multilayer design. Today's multilayer reflectivity's approach the theoretical limit and can hardly be improved further.

Thus, Liouville's theorem tells us that, given a particular source, the only way the optic can increase the intensity at the sample is by increasing the convergence solid angle.

It is thus desirable to have a large convergence angle in order to have a high intensity at the sample. However, it is of course possible to have too much convergence. This is due to the fact that, as the convergence angle increases, the size of the diffracted spots also increases (since the divergence of the diffracted beam is proportional to the convergence of the incident beam) as does the number of diffracted spots (since more reflections will be in Bragg resonance). Therefore, for a given unit cell length, there is a maximum tolerable beam convergence beyond which the spots begin to overlap, making integration difficult or impossible.

Because of this, modern optics are typically designed to have an adjustable beam converge. The optic should be designed for high convergence ratio to achieve high

intensity and then also incorporate adjustable apertures to allow the user to reduce the convergence angle when working with long unit cell samples.

This allows the user to achieve the maximum possible intensity for any sample through the proper selection of the limiting aperture.

As noted above, in Bruker diffraction equipment the Helios optics come with several preset apertures that allow the convergence angle to set from 7.6 mrad to 3 mrad. At 7.6 mrad, the corresponding convergence solid angle is  $\Omega=58 \mu\text{sr}$ . This is the highest convergence angle of any available multilayer optics and thus Helios gives the highest gain of any available multilayer optic. For example, the closest competitor to the Helios, the Rigaku VariMax HF only has a convergence angle of 4.8 mrad, corresponding to a convergence solid angle of 23  $\mu\text{sr}$ . Thus, on a given source, the Helios optics allow more than two times (58/23) higher intensity gain than the closest competing optics.

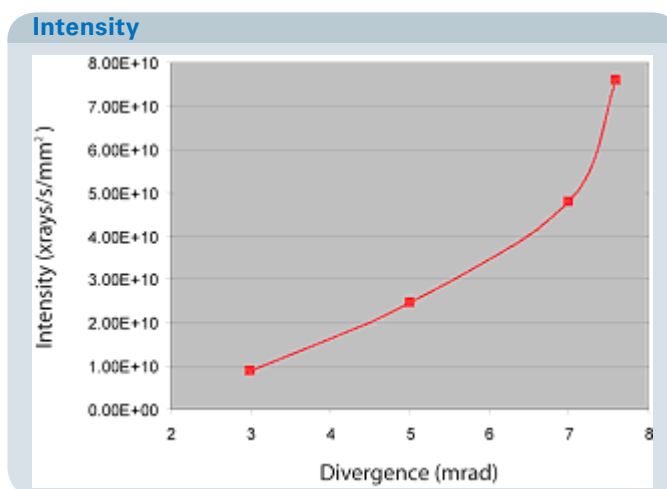


Figure 7: Intensity versus divergence for Helios optics (integrated with the MicroSTAR ULTRA source).

Using this largest convergence setting, the maximum unit cell that can be resolved is about 150 Angstroms. Other apertures allow larger unit cells to be resolved, up to about 450 Angstroms with the 3 mRad aperture (special apertures can be provided to allow even longer unit cells to be resolved).

Figure 7 shows the relation between flux through a typically sized crystal (0.1 mm) crystal and the d-spacing that can be resolved. Note the decreasing flux for large d-spacings, which is a consequence of Liouville's theorem.

Also shown in the figure is the occurrence of protein d-spacings (from 'The Protein Data Bank'). The distribution shows that macromolecular molecules require beams that

can resolve a wide range of d-spacings from less than 100 Å up to 400-500 Å. The Helios optic covers the whole range of relevant d-spacings.

The figure further shows the performance of the VariMax optic. The Helios outperforms the VariMax in the entire range. This is partly because the Helios produces a circular beam, which is more efficient than the square beam produced by the VariMax. The other reason is that it can produce a large convergence, making it also useful for medium- and small d-spacings. In this property the Helios is unique in the world.

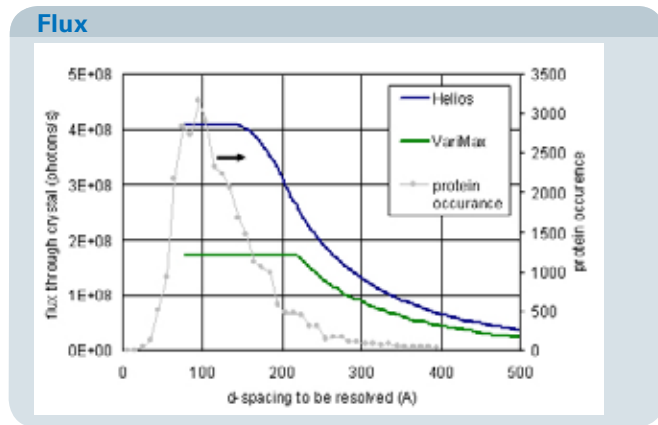


Figure 8: Flux through a 0.1 mm sized crystal as function of the d-spacing that is resolved, for two different optics (integrated with the Microstar).

A great advantage of the preset apertures used in the diffraction systems where the Helios is used, is that they are absolutely reproducible, unlike cross slits. In addition, as noted above, these apertures produce an ideal, truly circular beam profile, unlike the square beam profile produced by crossed slits.

### Optics Length and Intensity

It is often assumed that the size of the optic is directly proportional to its performance. However, this is not necessarily true. As noted above, the most important parameters for the optical performance are the convergence solid angle  $\Omega$  and the beam size  $D$  at the sample. An optic is designed such that these two properties meet the requirements of the diffraction experiment. As we will show below an optic with a large length does not necessarily result in better values for  $\Omega$  and  $D$ .

Figure 9 schematically shows the principle of a focusing multilayer optic. The mirror, with length  $L$ , sits on an ellipse with primary axes  $a$  and  $b$ . The source and sample sit in the ellipse foci at a distance  $f_1$  and  $f_2$  to the mirror middle. It then follows that  $\Omega$  and  $D$  are approximated by:

$$\Omega \approx \theta^2 \frac{L^2}{f_2^2}, \quad D \approx S \frac{f_2}{f_1},$$

in which  $\theta$  the diffraction angle of the multilayer and  $S$  the source size. Note that  $f_2/f_1$  is the magnification ratio of the optic. Multiplying this ratio with the source size gives the image (or beam) size at the sample.

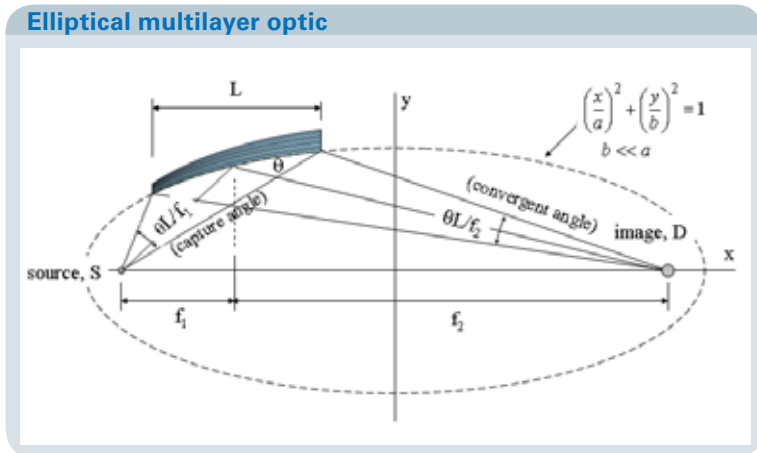


Figure 9: Schematic illustrating the working principles of an elliptical multilayer optic. The intensity gain of an optic depends on its length ( $L$ ) relative to its focal length ( $f_2$ ).

In the above equations,  $\theta$  and  $S$  are constants (multilayer for Helios:  $\theta=23$  mrad, and for Microstar:  $S=0.1$  mm). The design thus reduces to solving the two equations with three design parameters ( $L$ ,  $f_1$  and  $f_2$ ). This is usually done by setting  $f_1$  to a certain value, depending on how the design can be mechanically integrated with the source. The equations are then solved for  $L$  and  $f_2$ . The application for which the Helios is designed for (protein crystallography) requires  $\Omega=58$   $\mu$ str and  $D=200$   $\mu$ m, resulting in an optic with  $L=100$  mm and  $f_2=300$  mm.

The above shows the principles of how an optic is designed. These principles gives a rough estimate of the design and the optic properties. The final design is obtained by fine-tuning the initial design using advanced ray tracing simulations including detailed source and multilayer properties.

Although the above equations are only an approximation, they can be used to compare the performance of other optics with the Helios, like for example the VariMax. This optic is longer and has a larger focal length ( $L=120$  mm,  $f_2 \approx 500$  mm) than the Helios ( $L=100$  mm,  $f_2=300$  mm). The VariMax convergence angle is less than for the Helios, by more than a factor of 2. Consequently, the VariMax puts less flux onto the sample. In addition, there is the larger focal length  $f_2$  of the optic, resulting in a larger magnification ratio and thus a larger beam. The beam is thus too large and flux is wasted because it does not hit the crystal, only contributing to the noise and not to the signal.

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